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RELIABILITY OF ENERGY DISPERITIVE X-RAY FLUORESCENCE ANALYSIS OF LOW-ALLOY STEELS.

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POLYMER RESEARCH DIVISION

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ABSTRACT

The reliability of energy dispersive X-ray fluorescence data for the analysis of low-alloy steels is reported for chromium, manganese, nickel, and molybdenum. The accuracy and precision of the analysis were evaluated for two mathematical models, linear and multiple regression, using twelve NBS standard reference samples. Data on results are furnished.

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PREFACE

Three appendixes have been added as an addendum to this report. These were not included in the paper originally published due to space limitations. These appendixes provide information on instrument parameters, learn routine, and typical linear and multiple regression inputs.

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CONTENTS

	Page
PREFACE	iii
ABSTRACT	1
INTRODUCTION	1
EXPERIMENTAL	2
Linear Regression	2
Multiple Regression	3
RESULTS AND DISCUSSION	4
LITERATURE CITED	7
APPENDIX A. INSTRUMENT PARAMETERS AND LEARN ROUTINE	9
APPENDIX B. TYPICAL LINEAR REGRESSION INPUT AND COEFFICIENT OUTPUT.	10
APPENDIX C. TYPICAL MULTIPLE REGRESSION INPUT AND COEFFICIENT OUTPUT.	12

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**RELIABILITY OF ENERGY DISPERSIVE X-RAY FLUORESCENCE ANALYSIS OF
LOW-ALLOY STEELS**

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Army Materials and Mechanics Research Center

Watertown, Massachusetts 02172

ABSTRACT

The reliability of energy dispersive x-ray fluorescence data for the analysis of low-alloy steels is reported for chromium, manganese, nickel, and molybdenum. The accuracy and precision of the analysis were evaluated for two mathematical models, linear and multiple regression, using twelve NBS standard reference samples. Data on results are furnished.

INTRODUCTION

The widespread use of wavelength dispersive X-ray fluorescence spectrometry has been well documented in scientific journals for many years. All kinds of solids, liquids, and gases have been analyzed, and with very good accuracy. Recently, instruments based on energy dispersive X-ray fluorescence spectrometry have become available for analyzing the same types of materials; however, their major asset is a much more rapid analysis, with major and minor constituents immediately and simultaneously identified. Contrary to wavelength dispersive X-ray spectrometry not much has been documented on the accuracy and precision of the technique.

This paper describes our experience in determining chromium, manganese, nickel, and molybdenum in low-alloy steels and shows typical precision and accuracy data of this technique when using a Finnigan-900A spectrometer. This instrument, manufacturered by Finnigan Corp., Sunnyvale, CA, is equipped with an air-cooled rhodium target X-ray tube capable of 50 KV, 5 mA operation for primary X-ray excitation, the silicon detector, and a computer to automate operations and handle the data output.

Quantitative analysis may be automated by either a linear or a multiple regression program resident in the computer. For the low-alloy steels the linear regression was satisfactory for chromium, manganese, nickel, and molybdenum; however, when the chromium to manganese ratio exceeded 0.5 multiple regression was needed. As will be shown by the data, the multiple regression mode may be detrimental and should be used only when necessary.

Silicon, another important element in steel analysis, was examined but not successfully as the sensitivity of the detector was not adequate under optimum operating conditions for the other elements.

Through the use of a programmed or "LEARN" routine, standards of known composition were automatically excited for a pre-set period of time by radiation from the X-ray tube. Selected regions of the output spectrum were then examined for net area counts, with background automatically subtracted, and output data on intensity then registered. This output was then available for re-entry into the computer for either linear or multiple regression evaluation and eventual standardization of the instrument for the analysis of unknowns.

EXPERIMENTAL

All samples were prepared on a belt sander using an 80-grit aluminum oxide impregnated paper. Papers of 320- and 600-grit silicon carbide were then used, followed by final polishing with 3/0 emery paper. The samples in this study consisted of NBS 1161-68 and NBS 1261-64, which were used alternately as standards and unknowns. Chemical composition of these standards is given in Table I.

Basic parameters for the testing were: excitation from X-rays generated at 33.0 KV and 2.50 mA; 300-second acquisition time; and, pulse pile-up rejection (1) at approximately 25%. The peak regions selected for data acquisition and reduction were: 5.25 to 5.57 KEV for chromium; 5.78 to 6.03 KEV for manganese; 6.17 to 6.69 KEV for iron; 7.39 to 7.68 KEV for nickel, and 17.22 to 17.71 KEV for molybdenum. Background corrections were obtained from the region of 2.55 and 9.22 KEV for all elements except molybdenum, for which clear regions at 17.00 and 18.21 were chosen.

Linear Regression:

The twelve prepared standards were loaded onto the turntable and run automatically. For each element in the standard, the output net intensity (expressed as counts per second) and concentration were entered into the data system where a linear least squares fit was derived. The coefficients of that mathematical relationship were then outputted and available for future input when applicable.

TABLE I
NBS CERTIFICATION VALUES

SAMPLE NO.	Cr	Mn	Ni	Mo
1161	.13	.36	1.73	.30
1162	.74	.94	.70	.08
1163	.26	1.15	.39	.12
1164	.078	1.32	.14	.029
1165	.004	.032	.026	.005
1166	.011	.113	.051	.011
1167	.036	.28	.088	.021
1168	.54	.47	1.03	.20
1261	.69	.66	1.99	.19
1262	.30	1.04	.59	.068
1263	1.31	1.50	.32	.03
1264	.065	.26	.14	.49

The mathematical relationship is:

$$\% \text{ Element} = C_0 + C_1 I$$

where I is the intensity in counts per second and C_0 and C_1 the usual coefficients for a straight line relationship.

After the output of coefficients, unknowns were analyzed by simply placing them on the turntable and proceeding with the same "LEARN" routine as before.

Multiple Regression:

The multiple regression program includes a form of the Lucas-Tooth and Pyne equation (2). Under this program simultaneous equations are examined for best fit of coefficients to correct for inter-element effects (3). The same basic "LEARN" routine used for the linear regression (L.R.) is used to obtain data for input to set up the multiple regression (M.R.) analysis. An example of one of the equations, for manganese, used in this technique is:

$$\begin{aligned} \% \text{ Manganese} = & B_0 + B_1 I_{\text{Mn}} + B_2 I_{\text{Mn}}^2 + B_3 I_{\text{Mn}} I_{\text{Cr}} + B_4 I_{\text{Mn}} I_{\text{Fe}} \\ & + B_5 I_{\text{Mn}} I_{\text{Ni}} + B_6 I_{\text{Mn}} I_{\text{Mo}} \end{aligned}$$

where I is the intensity, in counts per second, of the appropriate element and B the coefficients derived from best-fit manipulation of the data.

The calculated B values are outputted on a teletype and on a punched tape. The punched tape is subsequently used to re-enter the coefficients for full automation of the analysis of unknowns.

RESULTS AND DISCUSSION

The first phase of the investigation was to determine the extent of reliability that was possible. By using all twelve standards to obtain the best possible linear and multiple regression coefficients, and by then using them as unknowns both precision and accuracy could be ascertained. Results of the standards as unknowns are shown in Table II for linear regression and in Table III for multiple regression. The average results reported is the average of 10 separate days of testing, each day's results being the average of 3 runs. Sigma (δ) values are for standard deviation on the ten results. For each element, standards are listed in descending order of concentration to aid in observing the limits of reliability.

The same data used to derive coefficients for the linear mode were used in obtaining the coefficients for the multiple regression, except that iron was included in the latter to accommodate for its interelement effect on all elements.

One can see that L.R. is reliable for the chromium and nickel down to about 0.05% and is exceptionally good to about 0.005% for molybdenum. Manganese, on the other hand, is most unsatisfactory by this mode and must be analyzed by the M.R. mode in order to obtain reasonable accuracy. However, to rely on M.R. for all elements would be at the expense of some accuracy for the others.

The manganese K_α peak is readily affected by the chromium K_β peak. Table IV shows the data of Tables II and III in terms of the effect of the chromium/manganese ratio on the manganese accuracy. The significant positive bias on the L.R. results persists down to a ratio of about 0.5. This does suggest that the L.R. can be used for all elements when that ratio is not exceeded. No explanation is offered for the results on the sample No. 1164.

The entire program was repeated and similar results were obtained, which confirmed the findings reported here.

TABLE II
ACCURACY AND PRECISION OF EXPERIMENTALLY DETERMINED VALUES USING LINEAR REGRESSION

CHROMIUM				MANGANESE			
SAMPLE	NBS	AVE.		SAMPLE	NBS	AVE.	
NO.	VALUE	EXP.	1σ	NO.	VALUE	EXP.	1σ
1263	1.31	1.31	.028	1263	1.50	1.59	.031
1162	.74	.76	.012	1164	1.32	1.22	.013
1261	.69	.71	.016	1163	1.15	1.07	.019
1168	.54	.56	.011	1262	1.04	.98	.025
1262	.30	.28	.011	1162	.94	.99	.037
1163	.26	.26	.012	1261	.66	.76	.021
1161	.13	.13	.014	1168	.47	.55	.013
1164	.078	.10	.009	1161	.36	.37	.014
1264	.065	.064	.010	1167	.28	.26	.011
1167	.036	.022	.006	1264	.26	.24	.010
1166	.011	.014	.005	1166	.113	.12	.012
1165	.004	.009	.007	1165	.032	.056	.009
NICKEL				MOLYBDENUM			
SAMPLE	NBS	AVE.		SAMPLE	NBS	AVE.	
NO.	VALUE	EXP.	1σ	NO.	VALUE	EXP.	1σ
1261	1.99	2.00	.039	1264	.49	.49	.006
1161	1.73	1.73	.031	1161	.30	.29	.004
1168	1.03	1.02	.014	1168	.20	.19	.004
1162	.70	.70	.013	1261	.19	.19	.003
1262	.59	.58	.020	1163	.12	.13	.007
1163	.39	.36	.028	1162	.080	.075	.003
1263	.32	.32	.013	1262	.068	.075	.004
1264	.14	.14	.007	1263	.030	.031	.003
1164	.14	.14	.014	1164	.029	.022	.002
1167	.088	.094	.009	1167	.021	.019	.004
1166	.051	.067	.010	1166	.011	.010	.003
1165	.026	.048	.013	1165	.005	.004	.003

TABLE III

ACCURACY AND PRECISION OF EXPERIMENTALLY DETERMINED VALUES USING MULTIPLE REGRESSION

CHROMIUM				MANGANESE			
SAMPLE NO.	NBS VALUE	AVE. EXP. VALUE	1σ	SAMPLE NO.	NBS VALUE	AVE. EXP. VALUE	1σ
1263	1.31	1.30	.035	1263	1.50	1.56	.072
1162	.74	.64	.045	1164	1.32	1.36	.039
1261	.69	.69	.020	1163	1.15	1.15	.043
1168	.54	.48	.034	1262	1.04	1.07	.046
1262	.30	.26	.007	1162	.94	.88	.041
1163	.26	.23	.017	1261	.66	.71	.037
1161	.13	.18	.027	1168	.47	.45	.022
1164	.078	.012	.010	1161	.36	.42	.024
1264	.065	.10	.035	1167	.28	.28	.017
1167	.036	.087	.023	1264	.26	.28	.022
1166	.011	.064	.004	1166	.113	.14	.007
1165	.004	.066	.004	1165	.032	.12	.008
NICKEL				MOLYBDENUM			
SAMPLE NO.	NBS VALUE	AVE. EXP. VALUE	1σ	SAMPLE NO.	NBS VALUE	AVE. EXP. VALUE	1σ
1261	1.99	1.95	.094	1264	.49	.51	.064
1161	1.73	1.70	.075	1161	.30	.32	.028
1168	1.03	.99	.045	1168	.20	.21	.021
1162	.70	.67	.043	1261	.19	.20	.015
1262	.59	.59	.030	1163	.12	.13	.012
1163	.39	.36	.024	1162	.080	.080	.008
1263	.32	.32	.018	1262	.068	.073	.008
1264	.14	.16	.029	1263	.030	.030	.003
1164	.14	.14	.0084	1164	.029	.030	.004
1167	.088	.10	.023	1167	.021	.028	.004
1166	.051	.017	.066	1166	.011	.019	.003
1165	.026	.049	.024	1165	.005	.011	.003

TABLE IV
EFFECT OF CHROMIUM ON MANGANESE VALUES

NBS SAMPLE NO.	Cr/Mn RATIO	NBS Mn	LINEAR		MULTIPLE	
			AMMRC	Diff.	AMMRC	Diff.
1168	1.149	.47	.55	+.08	.45	-.02
1261	1.045	.66	.76	+.10	.71	+.05
1263	.873	1.50	1.59	+.09	1.56	+.06
1162	.787	.94	.99	+.05	.88	-.06
1161	.361	.36	.37	+.01	.42	+.06
1262	.289	1.04	.98	-.06	1.07	+.03
1264	.250	.26	.24	-.02	.28	+.02
1163	.226	1.15	1.07	-.08	1.15	0
1167	.129	.28	.26	-.02	.28	0
1165	.125	.032	.036	+.004	.12	+.088
1166	.097	.113	.12	+.007	.14	-.027
1164	.059	1.32	1.22	-.10	1.36	+.04

In summary, the energy dispersive X-ray fluorescence technique provides a reliable and rapid method for determining chromium, manganese, nickel, and molybdenum in low-alloy steels. The dedicated computer to automate the operations permits the simultaneous determination of these elements, permits the choice of appropriate mathematical models to correct for interelement effects, and has the advantage of furnishing replication data for better statistics in a very short time frame.

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APPENDIX A. INSTRUMENT PARAMETERS AND LEARN ROUTINE

Parameters

33KV, 2.5mA, ACQUIRE TIME 300 sec, Pulse pile up rejection approx. 25% vertical full scale potentiometer was set at 16K. Gain 8, fine gain 0, baseline 88, post gain 1.

Learn Routine

The learn routine is executed by push button operation that sets up a program in the dedicated computer. Start/span positions varied very slightly on a day by day basis for the elements scanned and the entire work could be accomplished using the same start/span positions. This is only true if base line and fine gain potentiometers are set at constant value. The following Learn Routine was used:

Learn

First half

Acquire (data acquired for about a minute)

Acquire (stops acquiring)

Start Span select

Start/Span

Start/Span

Cr	40/8	230/8	121/9
Mn	40/8	230/8	136/7
Fe	40/8	230/8	147/15
Ni	40/8	230/8	182/8
Mo	454/3	488/4	462/14

Energy Ranges (KEV) for above Start/Span

Measured Energy Ranges

Element	Peak KEV	Scan Range	Bkg	Bkg
Cr	5.41	5.25-5.57	2.41-2.69	9.08-9.36
Mn	5.91	5.78-6.03	2.41-2.69	9.08-9.36
Fe	6.40	6.17-6.69	2.41-2.69	9.08-9.36
Ni	7.50	7.39-7.68	2.41-2.69	9.08-9.36
Mo	17.45	17.22-17.71	16.94-17.05	18.14-18.28

Calibration

Ti	CH. = 99.71	What Energy?	4.508 KEV
Co	CH. = 168.81	What Energy?	6.925 KEV
Mo	CH. = 468.46	What Energy?	17.443 KEV

$$A_0 = 1.0064 \quad A_1 = 0.0351$$

$$\text{Channel } (0.0351) + 1.0064 = \text{KEV}$$

Execute

Reset (May be used now so that a tape of the program can be obtained.)

Learn Tape (Tape is punched containing the program)

Execute (Instrument now proceeds with the routine)

APPENDIX B. TYPICAL LINEAR REGRESSION INPUT AND COEFFICIENT OUTPUT

Element Tag Cr

INT.	CONC.
13.96	1.31
2.66	.13
8.69	.74
3.83	.26
2.33	.078
1.26	.004
1.63	.011
1.41	.036
6.90	.54
8.60	.69
4.04	.30
2.12	.065

C

CO = - .13892E-00, C1 = .10153E-00

(C. C. = .998)

Element Tag Mn

INT.	CONC.
42.16	1.50
14.76	.36
28.45	.94
30.47	1.15
33.44	1.32
7.84	.032
9.25	.113
12.26	.275
18.13	.47
22.88	.66
28.04	1.04
12.43	.255

C

CO = .30813E-00, C1 = .45414E-01

(C. C. = .992)

Element Tag Fe

INT.	CONC.
2730	94.4
2917	96.5
2845	95.9
2871	96.0
2921	96.7
3006	99.4
3032	99.5
2911	97.5
2879	96.6
2849	95.6
2840	95.3
2907	96.7

C

CO = .43401E+02, C1 = .18415E-01

(C. C. = .995)

Element Tag Ni

INT.	CONC.
4.42	.32
22.69	1.73
9.41	.70
5.15	.39
2.10	.135
.90	.026
1.41	.051
1.51	.088
13.68	1.03
26.19	1.99
7.83	.59
2.07	.14

C

CO = -.29255E-01, C1 = .77456E-01

(C. C. = .9998)

Element Tag Mo

INT.	CONC.
4.21	.03
37.98	.30
11.08	.08
17.26	.12
3.37	.029
.47	.005
1.43	.011
2.51	.021
25.80	.20
24.66	.19
10.18	.068
64.47	.49

C

CO = -.14647E-02, C1 = .76766E-02

(C. C. = .9992)

APPENDIX C. TYPICAL MULTIPLE REGRESSION INPUT AND COEFFICIENT OUTPUT

Sample No. 1

Keyboard or Tape Input? K

No. of Samples = ? 12

No. of Elements = ? 5

Tag for element 00001? CR

Tag for element 00002? MN

Tag for element 00003? FE

Tag for element 00004? NI

Tag for element 00005? MO

INT = ?	CONC = ?
13.96 42.16 2730.74 4.42 4.21	1.31 1.50 94.4 .32 .03
2.66 14.76 2917.25 22.69 37.98	.15 .36 96.5 1.73 .30
8.69 28.45 2845.00 9.41 11.08	.74 .94 95.9 .70 .08
3.83 30.47 2871.08 5.15 17.26	.26 1.15 96.0 .39 .12
2.33 33.44 2921.23 2.10 3.37	.078 1.32 96.7 .135 .029
1.26 7.84 3006.75 .90 .47	.004 .032 99.4 .026 .005
1.63 9.25 3032.27 1.41 1.43	.011 .113 99.5 .051 .011
1.41 12.26 2911.20 1.51 2.51	.036 .275 97.5 .088 .021
6.90 18.13 2879.36 13.68 25.80	.54 .47 96.6 1.03 .20
8.60 22.88 2849.87 26.19 24.66	.69 .66 95.6 1.99 .19
4.04 28.04 2840.37 7.83 10.18	.30 1.04 95.3 .59 .068
2.12 12.43 2907.21 2.07 64.47	.065 .255 96.7 .14 .49

Turn on Punch; Hit Space.

B's for element CR.

.34551E-01 .38053E-00 .27745E-02-.21896E-03-.11669E-03 .65897E-03
-.14121E-03 .00000E+00 .00000E+00 .00000E+00 .00000E+00 .00000E+00

B's for element MN

.12322E-01 .12212E-00 .94603E-.1661E-02-.38065E-04 .31306E-03
.27249E-06 .00000E+00 .00000E+00 .00000E+00 .00000E+00 .00000E+00

B's for element FE

-.47043E-04 .50356E-01 .57376E-05 .43942E-04-.16778E-04-.13160E-04
-.40023E-05 .00000E+00 .00000E+00 .00000E+00 .00000E+00 .00000E+00

B's for element NI

.30450E-01 .41611E-00 .21683E-03-.62305E-03-.17185E-03-.12044E-03
.24471E-03 .00000E+00 .00000E+00 .00000E+00 .00000E+00 .00000E+00

B's for element MO

-.23523E-02-.23569E-01-.12202E-05 .10817-03-.16658E-04 .10747E-04
.76517E-05 .00000E+00 .00000E+00 .00000E+00 .00000E+00 .00000E+00

COEF. OR STD. INT.? C

STD. INT. OF CR = 13.96

STD. INT. OF MN = 42.16

STD. INT. OF FE = 2730.74

STD. INT. OF NI = 4.42

STD. INT. OF MO = 4.21

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